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Evaluation of a Muffle Furnace Procedure for Determining Percent Ash and Percent Weight Loss on Heating of Solid Wastes

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EVALUATION OF A MUFFLE FURNACE PROCEDURE FOR DETERMINING PERCENT ASH AND PERCENT WEIGHT LOSS ON HEATING OF SOLID WASTES

A Solid Waste Research
Open-File Report (RS-03-68-17)

written by
NANCY S. ULMER, Research Chemist

U.S. ENVIRONMENTAL PROTECTION AGENCY
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1971

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ABSTRACT

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INTRODUCTION

The population growth, urban sprawl, increased manufacture and sale of consumer goods, and the standard of living have been responsible for an ever-mounting increase in the mass and variety of materials being discarded in the United States. Scientists and engineers, engaged in the design, evaluation, and control of solid waste management systems, have depended upon the accurate determination of process parameters*, such as the quantity of heat released and the reduction in solid waste volume, weight, and volatile solids during incineration[1,2].

The determination of two parameters, the percent ash and percent volatile solids of refuse, incinerator residue and fly ash, and components thereof, have been performed by a variety of muffle furnace techniques, differing in procedural variations and conditions[3-12]. In fact, the determination of the percent volatile solids of solid wastes by methods, differing only in the muffle furnace temperature and crucible lid position, have resulted in the observation of not one, but two distinctly different parameters, namely, the weight lost by a sample in a closed crucible upon the volatilization of hydrocarbons, and the net weight lost by a sample in an open crucible upon the volatilization of the hydrocarbons, the combustion of the fixed carbon, and the oxidation (weight gain) of metals. Although the first observation of weight loss may be attributed to the loss of volatile solids alone, the second observation can not, and might best be presented simply as the "weight loss on heating".

Since the applicabilities of the various muffle furnace procedures have

*The term, parameter, is used herein to denote a variable or characteristic of interest.

not been established, an investigation was initiated in the Research Services Laboratory to determine the applicability of (1) the method tentatively proposed by the American Public Works Association[3] and (2) certain suggested or published variations in crucible preparation, sample preparation and weight, initial and final muffle furnace temperatures, rate and duration of sample heating, and crucible lid and furnace door positions in the determination of the percent weight loss on heating of solid wastes.

The approach, results, discussion, and conclusions of this investigation are presented in the following sections.

APPROACH

General

The feasibility of the tentative muffle furnace procedure was first evaluated by performing triplicate determinations of the percents ash and weight loss on heating of several solid waste samples. Since it became apparent immediately that the recommended crucible preparation, sample weight, and furnace door position were not always applicable or practical, independent studies were initiated to determine the applicabilities of specific variations in these and other procedural directions or conditions. Whenever a study indicated that a variation was more applicable or practical than the original technique, the variation was immediately adopted. Upon completion of all of the studies, the adopted variations were summarized and a modified procedure proposed. The accuracy, precision, and applicability of the modified procedure were then determined by analyzing a number of standard and solid waste samples.

Experimental

Equipment and supplies. A mechanically convected or forced-air oven, W-W Hammermill,* Iler Pulverizer, Wiley Laboratory Mill, and roller-mill and rotating-wheel-tumbling devices were employed in the preparation of solid waste samples. All analyses were performed using an analytical balance; seamless aluminum sample storage boxes, 3-1/2 inches in diameter; cabinet-type desiccator; Lindberg Hevi-Duty Muffle Furnace, Model 51333, with automatic Control Console, Model 59545; and high-form, Coors® porcelain crucibles (No. 2) with lids. The temperature of the furnace was monitored using a direct-reading, West Pyrotest, Model 9B, potentiometer with a chromel--alumel thermocouple and scale.

New crucibles and lids were first heated at 600 C for one hour in the muffle furnace in order to fix the applied Coors® ink numbers. After being used in a determination, they were cleaned with a non-metallic brush and detergent; rinsed thoroughly with tap water, and then distilled water; dried in a 110-C oven for one hour; and then stored in a desiccator until needed.

Reagents. Ground McDanel high-temperature, porcelain tube fragments were used as an inert standard. Strips of Whatman Filter Papers No. 41 (ashless) and No. 4, samples of analytical grade benzoic acid and sucrose, and three sucrose-porcelain tube fragment mixtures (3:1, 1:1, and 1:3 parts by weight) served as combustible standards. Analytical grade sodium carbonate and calcium carbonate were also employed in the investigation of the applicability of the procedure.

*Mention of commercial products does not imply endorsement by the U.S. Government.

Sample Preparation. Each compost and refuse sample was dried to constant weight in a 70- to 75-C oven; incinerator residue and fly ash were dried, however, at 100 to 105 C. After the particle diameter of a dried sample was reduced to 2 mm (or less) using a hammermill, pulverizer, or laboratory mill, the components were mixed in a one-gallon jar by tumbling on a roller mill or rotating wheel. A subsample (= 10 g) was then removed, transferred to an aluminum storage box, and redried for three hours at the previously specified temperature. After placing a tight-fitting lid on the storage box, it was transferred to a desiccator. The subsequent removal of small samples for analysis was always performed quickly in order to minimize the absorption of moisture by the stored sample.

American Public Works Association's Tentative Test for Ash and Volatile Solids. The published procedure directs the analyst to (1) transfer duplicate 3- to 6- g dried and ground solid waste subsamples to previously ignited and tared, high-form, Coors® porcelain crucibles, No. 1, 1A, or 2; (2) redry the samples for two hours at 75 C; (3) determine the sample weights to the nearest centigram; (4) introduce the crucibles into a cold muffle furnace; (5) gradually bring the furnace temperature to 600 to 650 C with the furnace door raised one-half inch; (6) expose the samples to this temperature for 2 hours; (7) transfer the crucibles to a desiccator; (8) determine the final sample weight and weight loss on heating after cooling; and (9) calculate the percents ash and volatile solids.

The technique, employed during the investigation of the published procedure, actually involved, however, the prior redrying of the samples, as described in the previous section; the triplicate determination of the parameters; the reading of all weights to the nearest 0.0001 g; the

exclusive use of the No. 2 crucibles; the covering of each crucible with a lid while weighing, tranporting, or cooling the crucible (or sample); the removal of the crucible lid during the heating of the sample in the muffle furnace; the heating of the furnace to a maximum temperature of 600 C; and the substitution of the term, weight loss on heating, for the term, volatile solids, in the presentation of all results.

Evaluation of Procedural Variations. The applicability of specific variations in the procedural techniques or conditions were evaluated by performing a number of determinations using, first, the tentative technique or condition and then each of the specified variations independently. To determine if the observations were characterized by homogeneity of variance (h.v.) and if there was any significant difference in the means (s.d.m.) of the observations, either a F test(h.v.) and a t test or a Cochran's C test and a F test(s.d.m.) and/or a t test were performed as described in Appendix A.

The following variations in procedural technique or condition were investigated as outlined:

1. crucible preparation -- omission of the pre-test ignition of each crucible-lid unit;
2. sample weight -- use of samples, each weighing approximately 2.0 g.
3. position of the furnace door during the gradual heat-up and exposure of samples to 600-C temperature -- maintenance of the furnace door in (a) a completely closed position or (b) a slightly open (3/16 inch) position;
4. position of the crucible lid during the gradual heat-up and 2-hour exposure of the samples to a 600-C temperature --maintenance of the

lid in (a) an "on" position or (b) a "tilted" position (with the angle of tilt being sufficient to permit the adequate circulation of air over the sample);

5. length of furnace heat-up period -- use of a 30- to 40-minute period to attain a 600-C temperature in the furnace (The original time required was 10 to 12 minutes);
6. initial furnace temperature -- use of an initial furnace temperature of 600 C (i.e., furnace pre-heated to 600 C before sample introduction);
7. maximum furnace temperature -- use of a 2-hour exposure of samples to a maximum furnace temperature of 500, 550, 650, 700, or 800 C;
8. length of exposure to maximum furnace temperature -- use of a 3- or 4-hour exposure of a sample at 600 C and a 1-hour exposure at 700 or 800 C;
9. sample preparation -- reduction of the solid waste particle diameter to (a) 1 mm or (b) 0.5 mm , and subsequent mixing;
10. number of determinations of each sample -- reduction of the number of determinations of each solid waste sample to two.

Evaluation of the Modified Procedure. The modified procedure was employed to determine the percent weight loss on heating (% WLOH) of standard and solid waste samples. The accuracy of the determinations of the standards was evaluated by comparing the theoretical % WLOH with the mean (M) of the % WLOH observations. The reproducibility of the observations of the standard and solid waste samples was determined by calculating the standard deviation (s) , the standard error ($s/\sqrt{2}$),

and the coefficient of variation (s/M) of each set of observations. Analyses of three types of waste components provided additional insight into the general applicability of the proposed modification.

RESULTS AND DISCUSSION

Applicability of the Tentative Procedure

Initial studies, in which the American Public Works Association's tentative procedure was utilized to determine the % WLOH of a refuse, a compost and a residue sample, immediately demonstrated that three of the procedural directions or conditions were either inapplicable or impractical.

First, it was observed that a 2-hour period was required to ignite and cool the crucibles and lids prior to their use in the analyses of samples. If this step could be eliminated from the test procedure, the total time required for the determination of the % WLOH could be reduced by one-third.

Secondly, the largest of the recommended crucible sizes (No. 2) would not contain the 3- to 6- g samples, suggested in the procedure. The inapplicability was particularly evident during the weighing of refuse and compost samples, which are extremely light (in density) and fluffy as compared to the fly ash and residue samples. Two-gram samples required little, if any, compaction and appeared more appropriate.

Thirdly, the recommendation that the furnace door remain open 1/2 inch during the heating of the samples seemed unnecessary and perhaps

even impractical. The design of the furnace, employed in this investigation permits the gases to evolve through the small space between the door and chamber linings and then up through the vent in the top casing. Moreover, the early failure (breaking) of the cables, supporting the door, may have indicated that a strain was being exerted on them while the door was maintained in this position.

Since the elimination or modification of these three procedural directions or conditions would greatly enhance the applicability of this method in the Research Services Laboratory, the investigation of the procedural variations was initiated without evaluating the precision and accuracy of the tentative method.

Applicability of Procedural Variations

Crucible-lid preparation. Four studies were performed to determine the effect of omitting the pre-test ignition of the crucible-lid units upon the % WLOH determinations. In each study the mean weight change in three previously used, but cleaned, dried, and empty crucible-lid units was determined after a 2-hour exposure at 600 C. The corresponding changes in % WLOH of solid waste samples were calculated by multiplying each mean weight change by 100 and then dividing each product by the initial sample weights of 1.5, 2.0, and 2.5 g. Since an evaluation of the calculated data revealed that the maximum change in the % WLOH of a 1.5- to 2.5-g solid waste sample would be 0.02 (Table 1), the pre-test ignition of each crucible-lid units was omitted in the rest of this investigation and in the final method recommended for use by other analysts.

TABLE 1

EFFECT OF OMITTING THE PRE-TEST IGNITION OF CRUCIBLE-LID UNITS ON SOLID WASTE DETERMINATIONS

STUDY #	CHANGE (C) IN CRUCIBLE-LID UNIT WEIGHT *		CALCULATED CHANGE IN % WLOH OF SOLID WASTE SAMPLES (100 C/W)*,†		
	Obs.	Mean	W=1.5	2.0	2.5
1	+0.0004 +0.0003 +0.0002	+0.0003	+0.02	+0.02	+0.01
2	-0.0005 0.0000 0.0000	-0.0002	-0.01	-0.01	-0.01
3	0.0000 +0.0003 +0.0003	+0.0002	+0.01	+0.01	+0.01
4	0.0000 0.0000 0.0000	0.0000	0.00	0.00	0.00

*All weights are expressed in grams

†W = initial solid waste sample weight

Sample weight. Four determinations of the % WLOH of an Alexandria (Virginia) refuse sample, a Johnson City (Tennessee) compost sample containing 6 % sewage sludge, and a New York City residue sample (RSL 68-204) were performed using 3 (or more)-g and 2-g sample weights. F tests(h.v.) and t tests were performed to compare the observations obtained using the two different sample weights. Since all the F and t values (Table 2) were less than the applicable critical values, it was concluded that there was no significant difference in the variances or means of the observations. The use of 2-g samples was, therefore, adopted immediately and also proposed in the final method.

TABLE 2

STATISTICAL ANALYSIS OF THE EFFECT OF SAMPLE WEIGHT ON SOLID WASTE DETERMINATIONS

Sample Type	Sample Source		Sample weight	No. of determinations	Mean % WLOH	Std. dev. (±)	F test (h.v.)*	t test†
Refuse	Alexandria Va.	A	(3-4g)	4	91.27	0.11		
		B	(2g)	4	91.30	0.12		
		A,B					1.19	+0.36
Compost with 6% sewage sludge	Johnson City, Tenn.	A	(3-3.75g)	4	62.17	0.69		
		B	(2g)	4	62.63	0.71		
		A,B					1.06	+0.93
Residue (RSL 68-204)	New York City	A	(3-4.5g)	4	32.91	0.49		
		B	(2g)	4	33.20	0.50		
		A,B					1.04	+0.83

*$F_{.95}$ (3,3) = 9.28

†$t_{.95}$ (2,6) = ±2.45

Position of furnace door during the gradual heat-up and exposure of samples to 600-C temperature. Initially the % WLOH determinations in a closed furnace (door position B) were to be compared with those determinations in a furnace whose door was opened 1/2 inch (position A). During the course of this study, however, the furnace temperature control settings had to be revised as the furnace elements aged and were replaced. To determine (and periodically check) the furnace control settings required to achieve specific temperatures, it became necessary to introduce into the furnace (through the space between the door and chamber linings) an independent thermocouple, attached to a West Pyrotest Model B potentiometer. As a result, the door was opened about 3/16 inch (position C). The design of this study was, therefore, revised to include an evaluation of the effect of this door position on the % WLOH determinations.

Three determinations of the % WLOH of a Delaware County (Pennsylvania) refuse sample (RSL 69-102) were performed using each of the three aforementioned door positions. Since the C and F(s.d.m.) tests yielded values (Table 3) which were less than the applicable critical values, there appeared to be no significant difference in the variances or the means of the observations. Thereafter (and, in the final proposed method) the closed door position was usually employed. When it was necessary to check or monitor the furnace temperature, door position C (opened 3/16 inch) was used. (No failure of the furnace door cables occurred during the remaining studies.)

Position of the crucible lid during the gradual heat-up and 2-hour exposure of the samples to 600-C temperature. Three determinations of the % WLOH of an Alexandria (Virginia) refuse sample and a New York City residue sample (RSL 68-204) were performed using three lid positions,

TABLE 3

STATISTICAL ANALYSIS OF THE EFFECT OF FURNACE DOOR POSITION ON REFUSE DETERMINATIONS

Door position	No. of determi-nations	Mean % WLOH	Std. dev. (±)	Cochran's C test*	F test (s.d.m.)†
A (Opened 1/2 inch)	3	85.59	0.30		
B (Closed)	3	84.97	0.12		
C (Opened 3/16 inch)	3	85.28	0.45		
A, B, C				0.66	2.81

*$C_{.95}$ (3,2) = 0.87

†$F_{.95}$ (2,6) = 5.14

A (off), B (tilted), and C (on). Since utilization of lid position C actually prevented the circulation of air over the sample being heated in a crucible, an overall statistical evaluation of the determinations by all three treatments seemed inappropriate. The data, obtained using each alternative lid position (B or C) was, therefore, compared only with the data obtained using the control lid position (A). A review of the F tests(h.v.) and the t tests (Table 4) demonstrates that only the variance and mean of the determinations. performed using lid position B (tilted) were consistently similar to the variance and mean of the determinations performed using lid position A (off).

Since the use of tilted lids did not produce a significant effect on the % WLOH determinations, it was continued thereafter. In the final method the analyst is advised to use tilted lids to minimize any possible loss of solid material from the crucible during an unexpectedly violent ignition of an unknown sample.

Length of the furnace heat-up period. After the completion of one-third of this investigation, the furnace manufacturer revised the wiring scheme of the heating elements in an effort to prolong the element life in this furnace model. The adoption of this new wiring scheme, however, lenthened the period of furnace heat-up and hence the total exposure of samples 18 to 28 minutes. Eight determinations of a New York City residue sample (RSL 68-204), containing considerable organic material, were, therefore, performed before and after the rewiring to determine the effect of increased heat-up period on the % WLOH observations. Since the F (h.v.) and t tests of the observations yielded values less than the applicable

TABLE 4

STATISTICAL ANALYSIS OF THE EFFECT OF CRUCIBLE LID POSITION ON REFUSE AND RESIDUE DETERMINATIONS

Sample Type	Source	Lid Position		No. of determinations	Mean % WLOH	Std. dev. (±)	F test (h.v.)*	t test
Refuse	Alexandria Va.	A	(Off)	3	91.31	0.14		
		B	(Tilted)	3	91.32	0.17		
		C	(On)	3	87.32	1.38		
		A,B					1.47	+0.08†
		A,C					97.16	-4.98‡
Residue (RSL-68-204)	New York City	A	(Off)	3	32.77	0.65		
		B	(Tilted)	3	33.31	0.32		
		C	(On)	3	30.57	0.47		
		A,B					4.12	+1.29†
		A,C					1.91	-4.81†

*$F_{.95}(2,2) = 19.0$

†$t_{.95}(2,4) = \pm 2.78$

‡$t_{.95}(2,3) = \pm 3.18$

critical values (Table 5), there appeared to be no significant differences in the variances or means of the determinations due to the additional heat-up period. The rest of the investigation was, therefore, performed using the new wiring scheme.

TABLE 5

STATISTICAL ANALYSIS OF THE EFFECT OF THE LENGTH OF FURNACE HEAT-UP PERIOD ON RESIDUE DETERMINATIONS

Heat-up Period	No. of determi-nations	Mean % WLOH	Std. dev. (±)	F test (h.v.)*	t test†
A (10 - 12 min.)	8	33.11	0.55		
B (30 - 40 min.)	8	32.92	0.34		
A,B				2.62	-0.51

*$F_{.95}(7,7) = 3.79$

†$t_{.95}(2,7) = \pm 2.36$

Initial furnace temperature--Use of an initial furnace temperature of 600 C. Since the adoption of the new wiring scheme increased the total time required to perform each determination 18 to 28 minutes, the use of a furnace, preheated to 600 C before sample introduction, was considered. Solid waste samples were transferred to crucibles and their respective weights determined while the furnace was heated to 600 C. Each of the crucible-lid units was then introduced one at a time into the pre-heated furnace. Since the rapid heating of the samples often resulted

in a more violent ignition, and a flame to which the arm of the analyst was exposed as he adjusted the lids, the evaluation of this procedural variation was terminated without determining the actual effect on the % WLOH.

Maximum furnace temperature. Since furnace temperatures, ranging from 550 to 1100 C, have been recommended or employed for the analysis of solid wastes or components thereof[3-13], an investigation of the effect of furnace temperature upon the % WLOH was initiated. A number of determinations of an Alexandria (Virginia) refuse sample, a Johnson City (Tennessee) compost sample containing 6% sewage sludge and a New York City residue sample (RSL 68-204) were performed using a 2-hour exposure at 500 C (residue only), 550, 600, 650, 700, and 800 C. The C, F(s.d.m.), and/or t tests were performed to determine if the variances and means of the observations at 500, 550, 650, 700, or 800 C differed significantly from those at 600 C.

The C tests of the refuse, compost, and residue determinations all produced values (Tables 6, 7, and 8) below the applicable critical values. The determinations of each solid waste, therefore, appeared to be characterized by homogeneity of variance. Three of the four t tests of the refuse observations, one of the t tests of the compost observations, but none of the t tests of the residue observations yielded values below the applicable critical values. Hence it was concluded that the mean of the determinations of (1) refuse at 550 C, (2) compost at 550, 700, and 800 C, and (3) residue at 550, 650, 700, and 800 C all differed significantly from the corresponding mean of the determinations at 600 C.

TABLE 6

STATISTICAL ANALYSIS OF THE EFFECT OF FURNACE TEMPERATURE ON REFUSE DETERMINATIONS

Max. furnace temperature*	No. of determi-nations	Mean % WLOH	Std. dev. (±)	Cochran's C test†	F test (s.d.m.)‡	t test§
A (600 C)	3	91.32	0.17			
B (550 C)	3	88.92	0.52			
C (650 C)	3	91.17	0.25			
D (700 C)	3	90.91	0.29			
E (800 C)	3	90.86	0.32			
A,B,C,D,E				0.49	26.12	
A,B						-8.86
A,C						-0.55
A,D						-1.51
A,E						-1.70

*The samples were exposed to the specified furnace temperature for two hours.

†$C_{.95}(5,2) = 0.68$

‡$F_{.95}(4,10) = 3.48$

§$t_{.95}(5,10) = \pm 2.97$

TABLE 7

STATISTICAL ANALYSIS OF THE EFFECT OF FURNACE TEMPERATURE ON COMPOST DETERMINATIONS

Max. furnace temperature*	No. of determi-nations	Mean % WLOH	Std. dev. (±)	Cochran's C test†	F test (s.d.m.)‡	t test§
A (600 C)	6	62.73	0.58			
B (550 C)	6	60.91	0.52			
C (650 C)	6	63.34	0.46			
D (700 C)	6	64.41	0.46			
E (800 c)	6	64.61	0.40			
A,B,C,D,E				0.28	56.44	
A,B						-4.57
A,C						+1.53
A,D						+4.22
A,E						+4.72

*The samples were exposed to the specified furnace temperatures for two hours.

†$C_{.95}$ (5,5) = 0.51

‡$F_{.95}$ (4,25) = 2.74

§$t_{.95}$ (5,25) = ±2.65

TABLE 8

STATISTICAL ANALYSIS OF THE EFFECT OF FURNACE TEMPERATURE ON RESIDUE DETERMINATIONS

Max. furnace temperature*	No. of determi-nations	Mean % WLOH	Std. dev. (±)	Cochran's C test†	F test (s.d.m.)‡	t test§
A (600 C)	8	33.11	0.55			
B (500 C)	8	30.93	0.61			
C (550 C)	8	31.80	0.22			
D (650 C)	8	34.34	0.43			
E (700 C)	8	34.80	0.17			
F (800 C)	8	35.13	0.22			
A,B,C,D,E,F				0.38	143.00	
A,B						-9.64
A,C						-3.97
A,D						+3.73
A,E						+5.12
A,F						+6.12

*The samples were exposed to the specified furnace temperature for two hours.

†$C_{.95}$ (6,7) = 0.40

‡$F_{.95}$ (5,42) = 2.45

§$t_{.95}$ (6,42) = ±2.67

Since the observations of the % WLOH of the compost and residue, but not of the refuse, were consistently higher at 700 or 800 C, an additional six determinations of the refuse were performed at 600, 700, and 800 C. The results did not differ, however, from those reported in Table 6.

Length of exposure to the maximum furnace temperature. Since exposure periods, varying from 10 minutes to 3 hours, have been recommended in the literature[3,6,9-12], an investigation was initiated to determine the effect of the length of the exposure period on the % WLOH determinations of the refuse, compost, and residue samples, employed in the previous evaluation of the effect of furnace temperature on observations. Nine determinations of the % WLOH of the residue were performed after 2-, 3-, and 4-hour exposures at 600 C and after a 1-hour exposure at 700 and 800 C. Six determinations of the % WLOH of both the refuse and the compost were also performed after a 2-hour exposure at 600 C and a 1-hour exposure at 700 and 800 C.

Since only the C values for the compost and residue observations were below the applicable critical values (Tables 9-12), it appeared that the residue determinations, performed after 2-, 3-, or 4-hour exposure at 600 C, and the residue and compost determinations, performed after a 2-hour exposure at 600 C and a 1-hour exposure at 700 and 800 C, were the only determinations characterized by homogeneity of variance.

The F test(s.d.m.) of the residue determinations at 600 C yielded an F value below the critical level. No significant difference appeared to exist, therefore, in the means of the observations made after a 2-, 3-, or 4-hour exposure at 600 C. The F and/or t values of the remaining sets of refuse, compost, and residue determinations were above the critical

TABLE 9

STATISTICAL ANALYSIS OF THE EFFECT OF LENGTH OF EXPOSURE AT 600 C ON RESIDUE DETERMINATIONS

Exposure period	No. of determi-nations	Mean % WLOH	Std. dev. (±)	Cochran's C test*	F test (s.d.m.)†
A (2 hrs)	9	33.16	0.53		
B (3 hrs)	9	33.19	0.54		
C (4 hrs)	9	32.91	0.43		
A,B,C				0.38	0.86

*$C_{.95}$ (3,8) = 0.63
†$F_{.95}$ (2,24) = 3.40

TABLE 10

STATISTICAL ANALYSIS OF THE EFFECT OF OTHER EXPOSURES CONDITIONS ON REFUSE DETERMINATIONS

Exposure condition	No. of determi-nations	Mean % WLOH	Std. dev. (±)	Cochran's C test*	t test
A (600 C, 2 hrs)	6	91.39	0.24		
B (700 C, 1 hr)	6	89.12	0.41		
C (800 C, 1 hr)	6	90.15	0.82		
A,B,C				0.75	
A,B					-11.71†
A,C					- 3.29‡

*$C_{.95}$ (3,5) = 0.71
†$t_{.95}$ (2,10) = ±2.23
‡$t_{.95}$ (2,7) = ±2.36

TABLE 11

STATISTICAL ANALYSIS OF THE EFFECT OF OTHER EXPOSURE CONDITIONS ON COMPOST DETERMINATIONS

Exposure condition	No. of determinations	Mean % WLOH	Std. dev. (±)	Cochran's C test*	F test (s.d.m.)†	t test‡
A (600 C, 2 hrs)	6	62.73	0.58			
B (700 C, 1 hr)	6	61.73	0.52			
C (800 C, 1 hr)	6	63.58	0.47			
A,B,C				0.41	18.68	
A,B						-3.31
A,C						+2.81

*$C_{.95}$ (3,5) = 0.71

†$F_{.95}$ (2,15) = 3.68

‡$t_{.95}$ (3,15) = ±2.44

TABLE 12

STATISTICAL ANALYSIS OF THE EFFECT OF OTHER EXPOSURE CONDITIONS ON RESIDUE DETERMINATIONS

Exposure condition	No. of determi-nations	Mean % WLOH	Std. dev. (±)	Cochran's C test*	F test (s.d.m.)†	t test‡
A (600 C, 2 hrs)	9	33.16	0.53			
B (700 C, 1 hr)	9	34.61	0.50			
C (800 C, 1 hr)	9	35.11	0.16			
A,B,C				0.51	49.62	
A,B						+4.10
A,C						+11.62

*$C_{.95}$ (3,8) = 0.63

†$F_{.95}$ (2,24) = 3.40

‡$t_{.95}$ (3,24) = ±2.35

values. The means of the determinations, performed after a 1-hour exposure at 700 or 800 C, appeared to differ significantly from those determinations, performed after a 2-hour exposure at 600 C. The review of the signs of the t tests further indicated that the means of the determinations of (1) the compost after a 1-hour exposure at 800 C and (2) the residue after a 1-hour exposure at 700 to 800 C were consistently larger than the corresponding means after a two-hour exposure at 600 C.

Until the nature and significance of the greater weight losses, observed in this and the previous study, can be evaluated more fully by the Research Services Laboratory Staff, the use of the 2-hour exposure at 600 C should be continued.

Sample Preparation. A study was performed to detect the effect of particle diameter and mixing upon the % WLOH observations. A Delaware County (Pennsylvania) refuse sample (RSL 69-102) was ground in a Wiley Mill using a 2-mm sieve. It was then mixed by tumbling on a 14-rpm rotary wheel. Half of this mixed sample (A) was reground in a Wiley Mill using a 1-mm sieve, and then tumbled as before. The resulting sample (B) was again divided into 2 portions. One of these portions was was reground in the mill using a 1/2-mm sieve. The final sample was tumbled as before and labelled sample C.

Three determinations of the % WLOH of each of three samples (A, B, and C) were performed using a 2-hour exposure at 600 C. Since the C and F(s.d.m.) tests produced values below the critical levels (Table 13) there appeared to be no significant difference in the variances or the means of the determinations. Additional evaluations should be performed, however, when other sample preparation techniques are employed.

TABLE 13

STATISTICAL ANALYSIS OF THE EFFECT OF
PARTICLE DIAMETER ON REFUSE DETERMINATIONS

Particle diameter	No. of determi-nations	Mean % WLOH	Std. dev. (±)	Cochran's C test*	F test (s.d.m.)†
A (2 mm)	3	85.24	1.38		
B (1 mm)	3	84.11	0.34		
C (0.5 mm)	3	84.76	0.83		
A, B, C,				0.70	1.08

*$C_{.95}$ (3,2) = 0.87
†$F_{.95}$ (2,6) = 5.14

Number of determinations. The effect of reducing the number of determinations of each solid waste from three to two was evaluated by performing triplicate determinations of the % WLOH of five refuse, eleven residue, and seven fly ash samples from Delaware County (Pennsylvania). The variance and mean of the three determinations of each sample were then compared with the variance and mean of the first two determinations of the sample. The F(h.v.) and t tests, performed on the refuse and residue observations (Tables 14 and 15), yielded values below the critical levels. The variances and means of the duplicate determinations of refuse and residue did not appear to differ significantly from the corresponding variances and means of the triplicate determinations.

A review of the data in Table 16 indicated that the standard deviations of the determinations of fly ash $\leq$ 0.04, the differences between the

TABLE 14

STATISTICAL ANALYSIS OF THE EFFECT OF REDUCTION IN NUMBER OF DETERMINATIONS ON REFUSE OBSERVATIONS

RSL sample #69-	No. of determinations A = 3, B = 2	Mean % WLOH	Std. dev. (±)	F test (h.v.)	t test*
99	A	87.60	0.07		
	B	87.58	0.08		
	A,B			1.31†	-0.29
100	A	89.06	0.09		
	B	89.03	0.10		
	A,B			1.23†	-0.36
101	A	86.25	0.30		
	B	86.08	0.06		
	A,B			25.00‡	-0.75
102	A	87.13	0.96		
	B	86.76	1.01		
	A,B			1.11†	-0.41
103	A	91.66	0.55		
	B	91.38	0.37		
	A,B			2.21‡	-0.40

*$t_{.95}(2,3) = \pm 3.18$

†$F_{.95}(1,2) = 18.5$

‡$F_{.95}(2,1) = 200$

TABLE 15

STATISTICAL ANALYSIS OF THE EFFECT OF REDUCTION IN NUMBER OF DETERMINATIONS ON RESIDUE OBSERVATIONS

Sample Fraction	RSL #69-	No. of determinations A = 3, B = 2	Mean % WLOH	Std. dev. (±)	F test (h.v.)	t test*
Combustible†	105	A	51.96	0.40		
		B	52.04	0.52		
		A,B			1.69‡	+0.20
Combustible	107	A	56.87	0.92		
		B	56.48	0.90		
		A,B			1.04§	-0.17
Combustible	109	A	63.88	1.66		
		B	62.96	0.65		
		A,B			6.52§	-0.71
Combustible	111	A	49.14	0.32		
		B	49.30	0.22		
		A,B			2.12§	+0.60
Combustible	113	A	62.13	1.66		
		B	61.80	2.21		
		A,B			1.77‡	-0.19

(Continued)

TABLE 15
(continued)

STATISTICAL ANALYSIS OF THE EFFECT OF REDUCTION IN NUMBER OF DETERMINATIONS ON RESIDUE OBSERVATIONS

Sample Fraction	RSL #69-	No. of determinations A = 3, B = 2	Mean % WLOH	Std. dev. (±)	F test (h.v.)	t test*
Fines¶	104	A	15.86	0.12		
		B	15.85	0.17		
		A,B			2.01‡	-0.08
Fines	106	A	12.55	0.29		
		B	12.63	0.37		
		A,B			1.63‡	+0.27
Fines	108	A	12.71	0.36		
		B	12.92	0.06		
		A,B			36.00§	+0.78
Fines	110	A	11.84	0.12		
		B	11.82	0.16		
		A,B			1.78‡	-0.16
Fines	112a	A	49.62	0.34		
		B	49.78	0.25		
		A,B			1.84§	+1.16
Fines	112b	A	25.30	0.66		
		B	25.63	0.47		
		A,B			1.97§	0.60

*$t_{.95}(2,3) = +3.18$

†The combustible fraction includes most of the residue that can be visually identified as containing food, paper, plastics, leather, wood, textiles, or garden wastes.

‡$F_{.95}(1,2) = 18.5$

§$F_{.95}(2,1) = 200$

¶The fines fraction includes all the unidentified and some of the identifiable residue particles that pass through a 0.5-inch wire mesh sieve.

TABLE 16

STATISTICAL ANALYSIS OF THE EFFECT OF REDUCTION IN NUMBER OF DETERMINATIONS ON FLY ASH OBSERVATIONS

RSL sample # 69-	No. of determi- A = 3, B = 2	Mean % WLOH	Std. dev. (±)
90	A	1.81	0.03
	B	1.80	0.02
91	A	5.04	0.03
	B	5.06	0.00
92	A	4.52	0.04
	B	4.51	0.04
93	A	3.83	0.03
	B	3.81	0.00
94	A	1.77	0.03
	B	1.78	0.03
96	A	2.75	0.01
	B	2.75	0.01
97	A	4.59	0.03
	B	4.58	0.02

standard deviations of the triplicate and duplicate determinations were $\leq$ .03, and the differences between the means of the triplicate and duplicate determinations were $\leq$ .02. Since none of these standard deviations or differences exceeded the practical limits suggested by analysts, the performance and averaging of duplicate fly ash determinations also seemed appropriate.

The number of determinations of the % WLOH of each sample was, therefore, reduced to two, assuming that (1) the Delaware County (Pennsylvania) samples were representative of those from other sources and (2) the data for two determinations and the data for three determinations are not interdependent. (Performance of five determinations of each sample would appear to have eliminated the necessity for the second assumption.)

Proposed Modification of the Tentative Procedure

The evaluation of the studies of the applicability of the tentative procedure and the procedural variations indicated that the crucible preparation, number of determinations, sample weight range, and furnace door position, recommended in the tentative procedure, were either inapplicable or unnecessary. It is, therefore, proposed that the method be modified by omitting the pre-test ignition of the crucible-lid units, reducing the number of determinations and the g-sample weight to two, and closing the furnace door completely or opening it only 3/16 inch. The modified procedural directions would then include:

(1) the prior preparation of the solid waste samples (i.e., drying at 70 or 105 C, particle-diameter reduction to 2 mm

or less, redrying at 70 or 105 C, mixing, and then storage in a desiccator);

(2) the transference of two 2-g subsamples of a solid waste to previously labelled, cleaned, dried, and weighed crucible-lid units (size No. 2);

(3) the determination of all weights to the nearest 0.0001 g;

(4) the introduction of the crucible-lid units, containing samples, into a cold muffle furnace;

(5) the tilting (or removal) of the crucible lids;

(6) the closure of the furnace door (except during the monitoring of the furnace temperature by an independent thermocouple);

(7) the gradual increase in furnace temperature to 600 C;

(8) a 2-hour exposure of the samples to the 600-C temperature;

(9) the subsequent transference and storage of the covered crucibles in a desiccator until they are cooled;

(10) and the determination of the final sample weight, weight loss, and % WLOH of each subsample and the mean % WLOH and mean % Ash of the solid waste.

Applicability of the Modified Procedure

Accuracy. An evaluation of the accuracy of the modified method was accomplished by performing duplicate determinations of two combustible standards, benzoic acid and sucrose; an inert standard (ground McDaniel-high-temperature-combustion-tube fragments); and three mixtures (3:1,

1:1, and 1:3 parts by weight) of the sucrose and the inert standard. The mean percents WLOH of benzoic acid, sucrose, and the three mixtures of sucrose and the inert standard were $\geq$ 99.92 percent of the theoretical percent WLOH (Table 17). Although the mean percent WLOH of the 3:1 mixture of sucrose and the inert standard was 100.45 percent of the theoretical percent WLOH, no weight loss was ever observed when the combustion tube fragments were heated alone.

TABLE 17

ACCURACY OF THE METHOD

Sample		Theoretical % WLOH (T)	Mean observed % WLOH (M)	(100 M/T)
Type	Identity			
Combustible standards	Benzoic acid	100	100.00	100.00
	Sucrose	100	100.00	100.00
	Sucrose-McDanel combustion tube, parts by weight,			
	3:1	75	75.34	100.45
	1:1	50	49.97	99.94
	1:3	25	24.98	99.92
Inert standard	McDanel combustion tube	0	0.00	

Reproducibility. Duplicate determinations of the aforementioned combustible and inert standards and the solid waste samples from Alexandria (Virginia), Johnson City (Tennessee), New York City, and Delaware County (Pennsylvania) were performed. The reproducibility of the modified

method was then determined by calculating the standard deviations of the observations, the standard errors of the means of the observations, and the coefficients of variation. A review of the observations and calculations (Table 18) indicates that the standard deviation of the determinations of the percents WLOH of the solid waste samples and the standard errors of the corresponding mean percents WLOH often exceeded and varied more than those of the standard samples. Since none of the coefficients of variation exceeded 0.04, however, the practical reproducibility requirements seemed to be satisfied. The nature or composition of other solid waste samples and the method of sample preparation may, of course, influence this reproducibility.

General applicability of the determinations of solid wastes and components thereof. The previous studies demonstrated the accuracy and precision of the determinations of combustible and inert standards and the precision of the solid waste determinations, but only the accuracy and precision of the determinations of one solid waste component, sucrose (a food component). The applicability of the procedure in the determination of the % WLOH of paper, the major solid waste component, had not been reported. Determinations of two types of paper, the Ashless Whatman filter paper No. 41 and the low-ash Whatman filter paper No. 4, were therefore performed. Since the mean percents WLOH of the observations were 100 and 99.96 respectively, and the standard deviations of the observations were $\leq$ .01 (Table 19), it was concluded that the method was applicable for the determination of the % WLOH of paper.

TABLE 18

REPRODUCIBILITY OF THE METHOD

Sample identity	RSL 69#-	Mean observed % WLOH (M)	Standard deviation of the determinations (±s)	Standard error of the mean (±s/√2)	Coefficient of variation (s/M)
Combustible standards:		100	0.00	0.00	0.00
Benzoic acid		100	0.00	0.00	0.00
Sucrose					
Sucrose-McDanel combustion tube, parts by weight, 3:1		75.34	0.06	0.04	0.00
1:1		49.97	0.14	0.10	0.00
1:3		24.98	0.01	0.01	0.00
Inert standard:					
McDanel combustion tube		0.00	0.00	0.00	-
Solid wastes:*					
Refuse	99	87.58	0.08	0.06	0.00
	100	89.03	0.10	0.07	0.00
	101	86.08	0.06	0.04	0.00
	102	86.76	1.01	0.71	0.01
	103	91.38	0.37	0.26	0.00
	AV	91.36	0.08	0.06	0.00
Residue, combustible fraction†	105	52.04	0.52	0.37	0.01
	107	56.48	0.90	0.64	0.02
	109	62.96	0.65	0.46	0.01
	111	49.30	0.22	0.16	0.00
	113	61.80	2.21	1.56	0.04

(Continued)

TABLE 18
(continued)

REPRODUCIBILITY OF THE METHOD

Sample identity	RSL #69-	Mean observed % WLOH (M)	Standard deviation of the determinations (±s)	Standard error of the mean (±s/√2)	Coefficient of variation (s/M)
Residue, fines fraction‡	104	15.85	0.17	0.12	0.01
	106	12.63	0.37	0.26	0.03
	108	12.92	0.06	0.04	0.00
	110	11.82	0.16	0.11	0.01
	112	25.63	0.47	0.33	0.02
	112a	49.78	0.25	0.18	0.01
Residue, non-graded	204	33.08	0.15	0.11	0.00
Fly Ash	90	1.80	0.02	0.01	0.01
	91	5.06	0.00	0.00	0.00
	92	4.51	0.04	0.03	0.01
	93	3.81	0.00	0.00	0.00
	94	1.78	0.03	0.03	0.02
	96	2.75	0.01	0.01	0.00
	97	4.58	0.02	0.01	0.00
Compost (with 6% sewage sludge)	JC	62.14	0.33	0.23	0.00

*The majority of the samples were collected at Incinerator No. 3, Delaware County, Pennsylvania. Sample "AV" is from Alexandria, Virginia; sample "JC" from Johnson City, Tennessee; and sample 204 from New York City.

†The combustible fraction includes most of the residue that can be visually identified as containing food, paper, plastics, rubber, leather, wood, textiles, or garden wastes.

‡The fines fraction includes all the unidentified and some of the identifiable residue particles that pass through a 0.5-inch wire mesh sieve.

TABLE 19

DETERMINATIONS OF PAPER

Paper type	No. of determinations	Mean % WLOH	Std. dev. (±)
Whatman ashless filter paper No. 41	3	100.00	0.00
Whatman low-ash filter paper No. 4	3	99.96	0.01

When other Research Services Laboratory studies demonstrated that some compost, residue, and fly ash samples contained significant quantities of carbonate[14], samples of Na_2CO_3 and $CaCO_3$ were also analyzed using this procedure. Since the observations (Table 20) revealed that only 0.12% of the Na_2CO_3 and 2.34 to 3.48 % of the $CaCO_3$ might be lost during a 2-hour exposure at 600 C, it appeared that the majority of the weight losses due to these components must occur at temperatures above 600 C and probably near the decomposition temperature of the carbonate (e.g., 960 C for the calcium carbonate form, calcite). The additional weight losses usually observed at 700 and 800 C during the (1) earlier evaluation of the procedural variations and (2) independent thermogravimetric studies (Appendix B) might, in fact, have been partially attributable to the weight losses of carbonate.

TABLE 20

CARBONATE DETERMINATIONS

Compound	No. of determinations	Mean % WLOH	Std. dev. (±)
Na_2CO_3	3	0.12	0.04
$CaCO_3$	3	2.34	0.38
$CaCO_3$ - benzoic acid (1:1 parts by weight)	3	3.48*	0.32

*Mean % WLOH of $CaCO_3$ was calculated assuming 100% WLOH of benzoic acid.

Since a knowledge of the possible effects of a partial or total decomposition of the carbonate components upon the mean % WLOH values would permit additional evaluation of the applicability of the modified furnace procedure, the carbonate determinations of a number of waste samples from Delaware County (Pennsylvania), Greenville (South Carolina), New Orleans, Johnson City (Tennessee), and Germany[14,15,16] were reviewed so that the samples with the highest carbonate levels could be selected from each group. The possible percents WLOH, attributable to a partial and a total decomposition of carbonate content, were then calculated assuming (1) that all the carbonates were present as $CaCO_3$ and (2) that 3.48% of the carbonate would decompose at 600 C.

A review of the calculated data (Table 21) revealed that the percents WLOH, attributable to the partial decomposition of $CaCO_3$ at 600 C, appeared to be small in all the selected samples except the New Orleans residue (1.00% WLOH) and possible the German compost (0.35% WLOH). If the selected samples were heated at a temperature $\geq$ 960 C (the highest of the decomposition temperatures of the forms of $CaCO_3$), the percent WLOH, attributable to the total decomposition of carbonate, would be $\geq$ 0.33% in all ten of the selected samples, $\geq$ 3.30% in six of the ten samples, and $\geq$ 10.13% in two of the ten samples.

Since the carbonate losses from solid wastes may be significantly higher at temperatures above 600 C, engineers and scientists may find it advisable to use % WLOH observations at 960 C (the highest of the decomposition temperatures of the common carbonates) to evaluate the efficiency of some solid waste reduction processes. Until additional research reveals the complete nature and significance of the other

TABLE 21

POSSIBLE PERCENT WEIGHT LOSSES ATTRIBUTABLE TO CARBONATE CONTENT OF SOLID WASTES*

Sample†		Mean %	Possible % WLOH#	
Source	Type	Carbonate Carbon	Partial Carbonate Decomposition	Total Carbonate Decomposition
Delaware Co., Pa.	Refuse	0.16	0.02	0.59
	Residue	0.93	0.12	3.41
	Fly Ash	0.15	0.02	0.55
Greenville, S.C.	Refuse	0.90	0.11	3.30
	Residue	0.40	0.05	1.49
New Orleans	Refuse (RSL 69-5)	0.09	0.01	0.33
	Residue (RSL 69-14)	7.91	1.00	29.02
	Fly Ash (RSL 69-22)	1.23	0.16	4.51
Johnson City, Tenn.	Compost	1.00§	0.13	3.67
Germany	Compost	2.76	0.35	10.13

*All carbonate present was assumed to be in the form $CaCO_3$.

†Only the samples with the highest carbonate carbon values are included here.

#The % WLOH was calculated assuming the volatilization of carbonate as CO_2.

§This is an approximate value.

weight losses and even gains (see thermogravimetric analyses, Figure B-6, Appendix B) that can occur in solid wastes at temperatures above 600 C, the general use of the modified procedure is recommended.

Relative Applicability of the Modified Procedure. Independent determinations of the % WLOH of New Orleans and Johnson City (Tennessee) solid wastes by the modified furnace procedure and thermogravimetric techniques (Appendix B) were briefly compared to determine the relative applicability of the furnace procedure. Only those thermogravimetric analyses (TGA) which employed air and yielded curves with plateaus at 600 and 1000 C, were used. Since an evaluation of the 600-C data indicated that the maximum difference in the % WLOH observations by the two methods was only 2.40 (Table 22), the methods appeared to have equal applicability at 600 C.

The significantly larger weight losses detected at 1000 C by the thermogravimetric procedures indicated again that some solid waste components evolve at temperatures above 600 C. When the probable percents weight loss, attributable to the total loss of the carbonate content as CO_2, were calculated (A in Table 23) and then subtracted from the corresponding actual percent weight loss occurring between 600 and 1000 C (B in Table 23), the resulting percents weight loss (B-A in Table 23) varied from -2.06 to +4.82. Evidently not all the solid waste weight changes occurring above 600 C could be attributed to a total loss of carbonate. The negative result obtained by subtracting the probable % WLOH of the residue sample (RSL 69-18) from the actual percent weight loss occurring above 600 C may indicate either that the carbonate decomposition was incomplete or partially masked by the oxidation (gain in weight) of another solid waste component. Until the nature and significance of all the possible solid waste weight

TABLE 22

COMPARISON OF THE DETERMINATIONS BY THE FURNACE AND THERMOGRAVIMETRIC PROCEDURES

Sample	% WLOH					% $WLOH_{TGA}$ - % $WLOH_{Furnace}$			
	Furnace	TGA (600 C)		TGA (1000 C)		600 C		1000 C	
		Dupont	Mettler	Dupont	Mettler				
	(A)	(B)	(C)	(D)	(E)	(B-A)	(C-A)	(D-A)	(E-A)
Refuse (RSL 69-5)	86.60	89.0	--	90.00	--	+2.40		+3.41	
Compost (JC-17G-D1)	86.54	--	84.0	--	--		-2.00		
Residue, organic fraction (RSL 69-14)	2.16	--	1.0	36.0	33.2	--	-1.16	+33.84	+31.04
Residue, organic fraction (RSL 69-18)	25.21	--	--	--	27.7	--	--	--	+2.49

TABLE 23

COMPARISON OF PROBABLE AND ACTUAL PERCENT WEIGHT LOSSES ABOVE 600 C

Sample	% Carbonate Carbon	Probable % WLOH* (A)	% $WLOH_{TGA(1000\ C)}$ - % $WLOH_{Furnace(600\ C)}$ (B)	(B-A)
Refuse (RSL 69-5)	0.69	0.33	3.41	+3.07
Residue, organic fraction (RSL 69-14)	7.91	29.02	33.84†	+4.82
			31.04†	+2.02
Residue, organic fraction (RSL 69-18)	1.24	4.55	2.49	-2.06

*Loss attributable to total loss of calcium carbonate as CO_2.
†% WLOH by duPont TGA.
†% WLOH by Mettler TGA.

changes above 600 C are investigated more thoroughly, a complete evaluation of the relative applicability of the modified furnace procedure can not be performed.

CONCLUSIONS

The initial investigation of the American Public Works Association's tentative procedure for the determination of the percent volatiles and ash suggested that the crucible preparation, sample-weight range, and furnace door position were either unnecessary or impractical. Evaluations of variations in these and seven other procedural variations or conditions revealed that the practical applicability of the tentative procedure could be enhanced by omitting the pre-test ignition of crucibles, reducing the sample weight to approximately two grams and the number of determinations to two, closing the furnace door (or, at most, opening it only 3/16 inch), and tilting the crucible lids during the exposure of samples to heat. The adoption of a modified method, incorporating these variations, and the substitution of the term, weight loss on heating, for the term, volatiles, was proposed. Additional studies then demonstrated that accurate and precise determinations of inert and combustible standards and a combustible solid waste (paper) and precise determinations of refuse, residue, fly ash, and compost could be obtained with this procedure. Since the percent weight loss of carbonate, another solid waste component, was low at 600 C, it was suggested that engineers and scientists might find it advisable to use % WLOH observations made at 960 C to evaluate the efficiency of some solid waste reduction processes. Additional studies of the nature and significance of the solid waste weight changes occurring above 600 C should be performed, however, before any modification in the maximum exposure temperature is proposed.

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ACKNOWLEDGMENTS

The efforts of Israel R. Cohen in preparing the solid waste samples and of Vence Lewis, Jr. in analyzing some of them are appreciated. Thanks are also extended to Donald L. Wilson for providing the carbonate data; to Paul W. Britton, George A. Garland, Daniel G. Greathouse, and Betty L. Grupenhoff for suggesting methods for the statistical evaluation of the observations; and to Jon P. Longtin (Division of Technical Support, Water Quality Office) for generously preparing the F test(s.d.m.)-computer program.

APPENDIX A

STATISTICAL EVALUATION OF THE EFFECTS OF THE VARIATIONS IN PROCEDURAL TECHNIQUE OR CONDITION ON PERCENT WLOH

The % WLOH data, collected during the analyses of subsamples of each solid waste by the control (tentative) and corresponding alternative treatments, was first tabulized. The mean % WLOH (M) and the standard deviation (s) of the determinations, made by each treatment, were then computed on the Olivetti Programma 101 using the following equations:

$$M = \frac{\Sigma x_{ij}}{n}$$

$$s = \sqrt{\frac{n_j \left(\Sigma x_{ij}^2\right) - \left(\Sigma x_{ij}\right)^2}{n_j^2 - n_j}}$$

where x_{ij} = an individual % WLOH observation by any treatment, j;

Σx_{ij} = the sum of individual % WLOH observations by any treatment, j;

and n_j = the number of observations by any treatment, j.

The determinations by the treatments were first tested for homogeneity of variance (h.v.) using either a F test(h.v.) or Cochran's C test. Any significant difference in the means (s.d.m.) of the determinations was then detected by applying a F test(s.d.m.) and/or t test(s). When k, the number of treatments being compared, was three (or more), Cochran's C test and a F test(s.d.m.) and/or t tests were employed. When the determinations by just the control and one alternative treatment (i.e., k = 2) were being evaluated, a F test(h.v.) and a t test were used. The general scheme for the statistical evaluation of the observations, the equations for calculating the F(h.v.), C, F(s.d.m.), and t tests and the applicable parameters of the sampling distribution, and the tables for determining the critical values at the

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95 percent confidence level are presented in the following pages. In practice the F test(s.d.m.) and the factor, MS_{error} [in the equation for t in approach (1) of the t test] were computed using the Olivetti Programma 101.

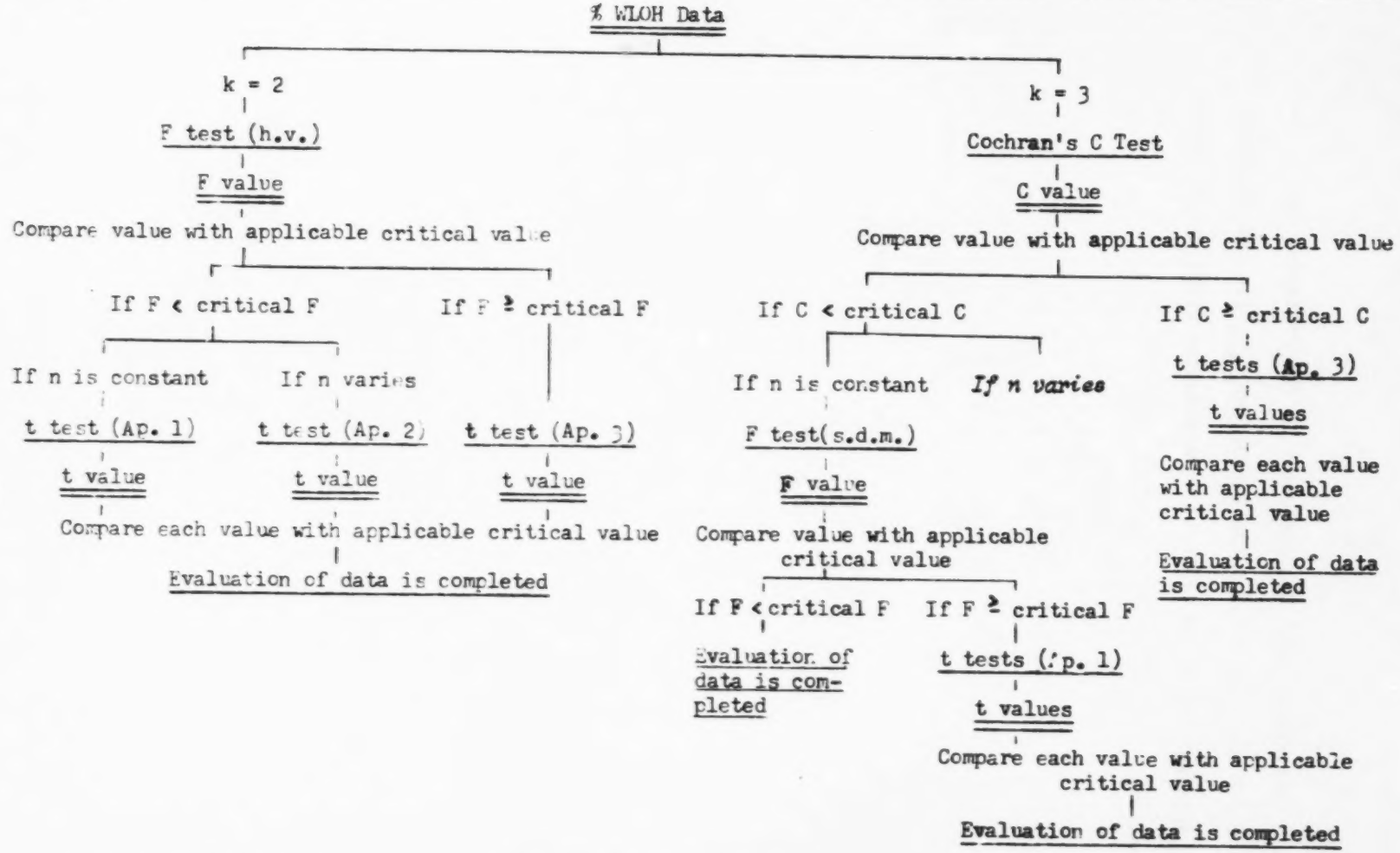
% WLOH Data
k = 2
F test (h.v.)
F value
Compare value with applicable critical value
If F < critical F
If F ≥ critical F
If n is constant
If n varies
t test (Ap. 1)
t test (Ap. 2)
t test (Ap. 3)
t value
t value
t value
Compare each value with applicable critical value
Evaluation of data is completed
k = 3
Cochran's C Test
C value
Compare value with applicable critical value
If C < critical C
If C ≥ critical C
If n is constant
If n varies
F test(s.d.m.)
F value
Compare value with applicable critical value
If F < critical F
If F ≥ critical F
Evaluation of data is completed
t tests (Ap. 1)
t values
Compare each value with applicable critical value
Evaluation of data is completed
t tests (Ap. 3)
t values
Compare each value with applicable critical value
Evaluation of data is completed

Figure A-1. Scheme for the statistical evaluation of the observed percent WLOH data, collected during the analyses of subsamples of a specific waste by the control (tentative) and alternative procedure(s). In the diagram k = the number of treatments, n = the number of determinations by a treatment, and the abbreviation, Ap., followed by a number, denotes the approach for calculating and interpretating the t value. The statistical approach for the italicized situation is not presented since it was not required in this study.

F test(h.v.)[1]

Purpose: To determine if the observations by two treatments, namely, the control (tentative) and an alternative procedure, are characterized by homogeneity of variance.

Calculation of F value:

$$F = \frac{s_{\ell}^2}{s_{s}^2}$$

Where s_{ℓ}^2 = the larger of the two variances;

s_{s}^2 = the smaller of the two variances.

Interpretation of the F(h.v.) test: From Table A-1 obtain the critical F value, $F_{1-\alpha,(n_{\ell}-1),(n_{s}-1)}$, at the 1-α confidence level using the sampling distribution parameters, $(n_{\ell}-1)$ and $(n_{s}-1)$,

where n_{ℓ} = the number of determinations, displaying the larger variance;

n_{s} = the number of determinations, displaying the smaller variance;

$n_{\ell}-1$ = the degrees of freedom (df) of the larger variance. i.e., the numerator of the F equation;

$n_{s}-1$ = the degrees of freedom (df) of the smaller variance, i.e., the denominator of the F equation.

If the magnitude of the calculated F value is less than the critical F value. then the observations are characterized by homogeneity of variance. If, however, the calculated F value ≥ the critical F value, the observations are not so characterized.

TABLE A-1

F DISTRIBUTION*
(1-α = 0.95)

df for denominator	df for numerator						
	1	2	3	4	5	6	7
1	161	200	216	225	230	234	237
2	18.5	19.0	19.2	19.2	19.3	19.3	19.4
3	10.1	9.55	9.28	9.12	9.10	8.94	8.89
4	7.71	6.94	6.59	6.39	6.26	6.16	6.09
5	6.61	5.79	5.41	5.19	5.05	4.95	4.88
6	5.99	5.14	4.76	4.53	4.39	4.28	4.21
7	5.59	4.74	4.35	4.12	3.97	3.87	3.79
8	5.32	4.46	4.07	3.84	3.69	3.58	3.50
9	5.12	4.26	3.86	3.63	3.48	3.37	3.29
10	4.96	4.10	3.71	3.48	3.33	3.22	3.14
11	4.84	3.98	3.59	3.36	3.20	3.09	3.01
12	4.75	3.89	3.49	3.26	3.11	3.00	2.91
13	4.67	3.81	3.41	3.18	3.03	2.92	2.83
14	4.60	3.74	3.34	3.11	2.96	2.85	2.76
15	4.54	3.68	3.29	3.06	2.90	2.79	2.71
16	4.49	3.63	3.24	3.01	2.85	2.74	2.66
17	4.45	3.59	3.20	2.96	2.81	2.70	2.61

(Continued)

TABLE A-1 (continued)

F DISTRIBUTION
($1-\alpha = 0.95$)

df for denominator	df for numerator						
	1	2	3	4	5	6	7
18	4.41	3.55	3.16	2.93	2.77	2.66	2.58
19	4.38	3.52	3.13	2.90	2.74	2.63	2.54
20	4.35	3.49	3.10	2.87	2.71	2.60	2.51
22	4.30	3.44	3.05	2.82	2.66	2.55	2.46
24	4.26	3.40	3.01	2.78	2.62	2.51	2.42
26	4.23	3.37	2.98	2.74	2.59	2.47	2.39
28	4.20	3.34	2.95	2.71	2.56	2.45	2.36
30	4.17	3.32	2.92	2.69	2.53	2.42	2.33
40	4.08	3.23	2.84	2.61	2.45	2.34	2.25
60	4.00	3.15	2.76	2.53	2.37	2.25	2.17

*Adapted from B. J. Winer (2) and E. S. Pearson and H. O. Hartley, eds. (7).

Cochran's C test[4]

Purpose: To determine if the observations by the control (tentative) and two (or more) alternative treatments are characterized by homogeneity of variance.

Calculation of C value:

$$C = \frac{s_\ell^2}{\sum_{j=1}^{k} s_j^2}$$

Where s_j = the standard deviation of the determinations by an individual treatment, j;

s_j^2 = the variance of the determinations by an individual treatment, j;

k = the number of variances or treatments;

and s_ℓ^2 = the largest of the k variances.

Interpretation of the C test: From Table A-2 obtain the critical C value, $C_{1-\alpha, [k, (n-1)]}$, at the 1-α confidence level using the sampling distribution parameters, k and (n-1),

where 1-α = 0.95;

k = the number of variances;

n = the number of determinations common to each treatment;

and n-1 = the degrees of freedom (df) common to each variance.

If the magnitude of the calculated C value is less than the critical C value, then the observations are characterized by homogeneity of variance. If, however, the calculated C value ≥ the critical C value, the observations are not so characterized.

TABLE A-2

CRITICAL VALUES FOR COCHRAN'S TEST FOR HOMOGENEITY OF VARIANCE*
($1-\alpha = 0.95$)

df = (n-1)	k = number of variances				
	2	3	4	5	6
1	.9985	.9669	.9065	.8412	.7808
2	.9750	.8709	.7679	.6838	.6161
3	.9392	.7977	.6841	.5981	.5321
4	.9057	.7457	.6287	.5441	.4803
5	.8772	.7071	.5895	.5065	.4447
6	.8534	.6771	.5598	.4783	.4184
7	.8332	.6530	.5365	.4564	.3980
8	.8159	.6333	.5175	.4387	.3187
9	.8010	.6167	.5017	.4241	.3682

*Adapted from B. J. Winer (5) and C. Eisenhart, M. H. Hastay, and W. A. Wallis (6).

F test(s.d.m.)[7]

Purpose: When the determinations by three (or more) treatments are characterized by homogeneity of variance, this test is employed to determine if there is any significant difference in the means of the determinations by the treatments.

Calculation of F value: When the number of determinations by each treatment is constant in three (or more) treatments being compared, the following equation is used to obtain the F value.

$$F = \frac{MS_{treatments}}{MS_{error}}$$

$$= \frac{\frac{SS_{treatments}}{k-1}}{\frac{SS_{error}}{k(n_j-1)}}$$

$$= \frac{\frac{\Sigma T_j^2}{n_j} - \frac{(\Sigma T_j)^2}{kn_j}}{\frac{\Sigma\Sigma x_{ij}^2 - \frac{\Sigma T_j^2}{n_j}}{k(n_j-1)}}$$

Where n_j = the number of observations or determinations by any treatment, j;

k = the number of treatments or variances;

x_{ij} = an individual observation or determination by any treatment, j;

$T_j = \sum_{i=1}^{n_j} x_{ij}$, the sum of the observations or determinations by any treatment, j;

$$\Sigma T_j = \sum_{j=1}^{k} \left(\sum_{i=1}^{n_j} x_{ij} \right) ;$$

and $$\Sigma\Sigma x_{ij}^2 = \sum_{j=1}^{k} \left(\sum_{i=1}^{n_j} x_{ij}^2 \right) .$$

Interpretation of the F test: From Table A-1 obtain the critical F value, $F_{1-\alpha,}$ [(k-1), k(n-1)], at the 1-α confidence level using the sampling distribution parameters, (k-1) and k(n-1),

where $1-\alpha$ = 0.95;

k = the number of means or treatments;

k-1 = the degrees of freedom (df) of $MS_{treatments}$, i.e., the numerator of the F equation;

n = the number of determinations common to each treatment;

and k(n-1) = the degrees of freedom (df) of MS_{error}, i.e., the denominator of the F equation.

If the magnitude of the calculated F value is less than that of the critical value, there is no significant difference in the means of the determinations. If, however, the calculated F value ≥ the critical value, then two (or more) t tests must be performed to determine which mean(s) of the determinations by the alternative treatments differ significantly from the mean of the determinations by the control (tentative) treatment.

t test

Purpose: To determine if there is any significant difference in the means of the determinations by two treatments, namely, the control (tentative) and an alternative procedure.

Calculation and interpretation of the t value: As outlined in Table A-3, one of three approaches is used depending upon the results of the F(h.v.) or Cochran's C test and on the equality of n_1 and n_2, the number of determinations by the control (tentative) and alternative treatments, respectively.

TABLE A-3

GUIDELINE FOR CHOOSING APPLICABLE APPROACH

F(h.v.) or Cochran's C test results: F or C value less than critical value (homogeneity of variance)	$n_1 = n_2$	Applicable approach
Yes	Yes	(1)
Yes	No	(2)
No	Yes or No	(3)

Approach (1) for calculating and interpretating the t value[8]:

$$t = \frac{\bar{x}_2 - \bar{x}_1}{\sqrt{\frac{2(MS_{error})}{n_j}}}$$

$$= \frac{\bar{x}_2 - \bar{x}_1}{\sqrt{\frac{2\left[\Sigma\Sigma x_{ij}^2 - \frac{\Sigma T_j^2}{n_j}\right]}{n_j[k(n_j-1)]}}}$$

Where x_{ij} = an individual observation or determination by any treatment, j;

T_j = the sum of the individual observations or determinations by any treatment, j;

$\bar{x}_1$ = the mean of the observations by the control (tentative) method;

$\bar{x}_2$ = the mean of the observations by the alternative treatment;

n_j = the number of determinations by any treatment, j;

k = the number of means (includes mean of the control and each alternative treatment);

and $\Sigma T_j^2 = \sum_{j=1}^{k} \left(\sum_{i=1}^{n_j} x_{ij} \right)^2$

When more than one alternative treatment is to be compared with the control treatment, k exceeds 2 and MS_{error} is calculated using the observations by all treatments.

The critical t value, $t_{1-\alpha,}$ [k, k(n-1)],is obtained from Table A-4 at the 1-α confidence level using the sampling distribution parameters, k and k(n-1),

where 1-α = 0.95

k = the total number of means;

n = the number of determinations common to each treatment;

and k(n-1) = the degrees of freedom (df) of the error variance, i.e., MS_{error}.

This table has been constructed using the Student's t distribution[9] when the degrees of freedom of error variance ≤ 4 and the Dunnett's t distri-

bution[10] when the degrees of freedom of error variance > 4. Dunnett prepared his table for use when α, the level of significance, is desired for the set of all the control and alternative method comparisons. When k = 2, the two distributions are equal. When k is greater than 2, the values in Dunnett's table are larger than those in the Student's t distribution table.

If the magnitude of the calculated t value is less than that of the critical t value, there is no significant difference in the means of the determinations by the control and the alternative methods. If, however, the calculated t $\geq$ the critical value, then there is a significant difference in the two means.

Approach (2) for calculating and interpretating the t value[11]:

$$t = \frac{\bar{x}_2 - \bar{x}_1}{\sqrt{\frac{2(MS_{error})}{\tilde{n}}}}$$

$$= \frac{\bar{x}_2 - \bar{x}_1}{\sqrt{\frac{2\left[\Sigma\Sigma x_{ij}^2 - \Sigma\left(\frac{T_1^2}{n_1} + \frac{T_2^2}{n_2} \ldots + \frac{T_k^2}{n_k}\right)\right]}{\tilde{n}\,(N-k)}}}$$

Where x_{ij} = an individual observation or determination by any treatment, j;

$\bar{x}_1$ = the mean of the observations or determinations by the first (or control) treatment;

$\bar{x}_2$ = the mean of the observations or determinations by a second (alternative) treatment;

T_1 = the sum of the observations by the first (control) treatment;

T_2 = the sum of the observations by a second (alternative) treatment;

T_k = the sum of the observations by treatment no. k;

k = the number of means or treatments;

n_1 = the number of determinations by the first (control) treatment;

n_2 = the number of determinations by the second (alternative) treatment;

n_k = the number of determinations by treatment no. k;

N = $n_1 + n_2 \ldots + n_k$;

and $\tilde{n} = \dfrac{k}{[\,1/n_1 + 1/n_2 \ldots + 1/n_k]}$

In the determination of the t value by the above equation, the calculated value of $\tilde{n}$ is utilized.

The critical t value, $t_{1-\alpha\,,\,[k,(N-k)]}$, is obtained from Table A-4 at the 1-α confidence level using the sampling distribution parameters, k and (N-k),

Where 1-α = 0.95;

k = the number of means;

and N-k = the degrees of freedom (df) of the error variance, i.e., MS_{error}.

The t test is interpretated by comparing the calculated and critical t values in the previously described manner.

Approach (3) for calculating and interpretating the t value[12]:

$$t = \frac{\bar{x}_2 - \bar{x}_1}{s_{\bar{x}_2 - \bar{x}_1}}$$

$$= \frac{\bar{x}_2 - \bar{x}_1}{\sqrt{\frac{s_{x_1}^2}{n_1} + \frac{s_{x_2}^2}{n_2}}}$$

Where $\bar{x}_1$ = the mean of the observations or determinations by the control treatment;

$\bar{x}_2$ = the mean of the observations or determinations by the alternative treatment;

$s_{x_1}^2$ = the variance of the observations by the control treatment;

$s_{x_2}^2$ = the variance of the observations by the alternative treatment;

n_1 = the number of observations by the control treatment;

n_2 = the number of observations by the alternative treatment.

The critical t value, $t_{1-\alpha,}(k, df)$, is obtained from Table A-4 at the $1-\alpha$ confidence level using the sampling distribution parameters, k and df

Where $1-\alpha$ = 0.95;

k = the number of means;

and df = the degrees of freedom of the error variance

$$= \frac{\left[\frac{s_{x_1}^2}{n_1} + \frac{s_{x_2}^2}{n_2}\right]^2}{\frac{\left[\frac{s_{x_1}^2}{n_1}\right]^2}{n_1+1} + \frac{\left[\frac{s_{x_2}^2}{n_2}\right]^2}{n_2+1}} - 2$$

In practice the next higher integer above the calculated df value was employed.

The t test is interpretated by comparing the calculated and critical t values in the previously described manner. Note that this approach provides an independent comparison of the two means since it utilizes only the variances of the observations of the two treatments being compared.

TABLE A-4

TWO-TAILED t DISTRIBUTION*
($1-\alpha = 0.95$)

df for variance error	k = number of means				
	2	3	4	5	6
1	12.71				
2	4.30				
3	3.18				
4	2.78				
5	2.57	3.03	3.39	3.66	3.88
6	2.45	2.86	3.18	3.41	3.60
7	2.36	2.75	3.04	3.24	3.41
8	2.31	2.67	2.94	3.13	3.28
9	2.26	2.61	2.86	3.04	3.18
10	2.23	2.57	2.81	2.97	3.11
11	2.20	2.53	2.76	2.92	3.05
12	2.18	2.50	2.72	2.88	3.00
13	2.16	2.48	2.69	2.84	2.96
14	2.14	2.46	2.67	2.81	2.93
16	2.12	2.42	2.63	2.77	2.88
18	2.10	2.40	2.59	2.73	2.84
20	2.09	2.38	2.57	2.70	2.81
24	2.06	2.35	2.53	2.66	2.76
30	2.04	2.32	2.50	2.62	2.72
40	2.02	2.29	2.47	2.58	2.67
60	2.00	2.27	2.43	2.55	2.63

*Adapted from R. A. Fisher and F. Yates (10) and from C. W. Dunnett (11).

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APPENDIX B

THERMOGRAVIMETRIC ANALYSES OF SOLID WASTES

During the course of the evaluation of the applicability of the tentative and modified muffle furnace procedures, thermogravimetric analyses of six solid waste samples were performed by representatives of E. I. duPont de Nemours, Mettler Instrument Corporation, and Robert L. Stone Company (Columbia Scientific Industries). The samples included a Johnson City (Tennessee) compost (JC-17G-D1); and five New Orleans solid wastes, a refuse (RSL 69-5), the organic fraction of two residues (RSL 69-14 and RSL 69-18), a fines fraction of a residue (RSL 69-13), and a fly ash (RSL 69-22).

The duPont representative used the duPont Thermal Analysis System 900 with the thermogravimetric module 950, a high temperature furnace, a platinum pan-type sample holder with a 200-mg capacity, a quartz rod support, a platinum —13 percent platinum rhodium furnace thermocouple, a chromel - alumel sample thermocouple, and a dynamic air atmosphere. The atomospheric flow rate, the sample heating rate, the chart speed, and the sample weight varied with the analysis. (See Figures B:1-6.)

The Mettler representative employed the Mettler Thermoanalyzer Model 2 with a quartz furnace, a platinum crucible, a platinum— 10 percent platinum rhodium thermocouple, a macro-DTA sample holder, and a dynamic air atmosphere for the analyses of the refuse, compost, and one residue (RSL 69-14). A high temperature furnace and a micro-DTA (TD-2) sample holder were employed for the analyses of the fly ash and other residue samples. In each analysis the chart speed was 12 inches per hour but the atmospheric flow rate, sample weight, and the heating rate varied with the sample. (See Figures B-1 and B-3 through B-7.)

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The Stone representative utilized the Stone Instrument, Model TGA-3C, with a platinum—platinel II thermocouple and a cylindrical nickel sample holder with a porous base disc which permitted a dynamic atmosphere flow through and surrounding the sample. In each analysis helium was introduced at a flow rate of 1.41 liters per hour. Since each sample was heated at 10 degrees (C) per minute and the chart width represented 1100 degrees (C), the chart speed was 120 degrees (C) per inch or 5.6 inches per hour. The sample size varied with the analyses.

The TGA curves, obtained for each sample, were replotted using the same axes and scale (Figures B; 1-7). All interpretations, comparisons, and other uses of the curves (or the % WLOH data derived therefrom) were made with the knowledge that the sample weight, atmosphere type and flow rate, and the heating rate may affect the observed % WLOH at any one specificed temperature.

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Thanks are also extended to Israel Cohen for preparing the solid waste samples and to Don Wilson for providing the carbonate data for the refuse, residue, and fly ash samples.

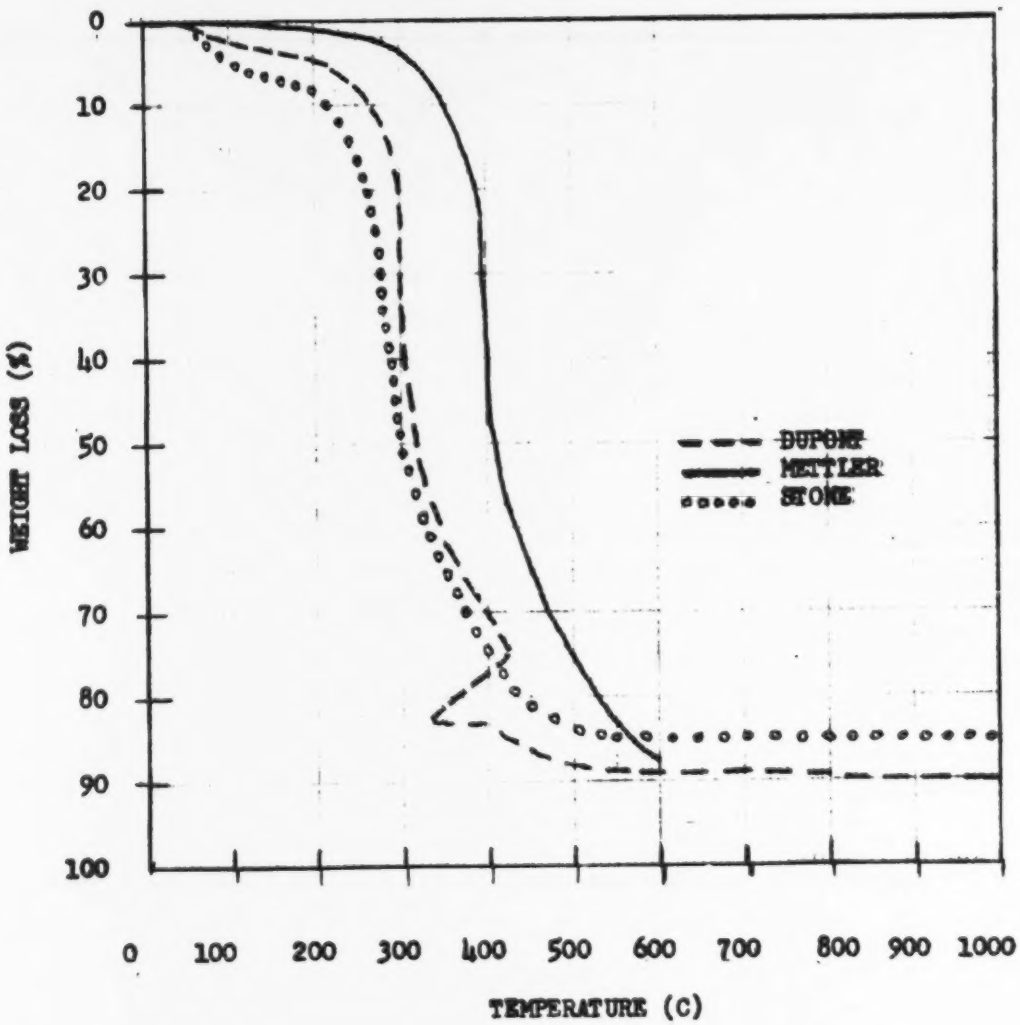

Figure B-1. Percent weight-loss curves of a municipal refuse (RSL 69-5). The curves were derived from thermogravimetric analyses performed under the following experimental conditions:

	duPont	Mettler	Stone
Sample weight (mg)	9.3	7.08	100
Atmosphere	Air	Air	Helium
Flow rate (liters/hr)	6	6	1.41
Heating rate (°C/min)	30	15	10
Chart speed (in./hr)	18	12	5.6

The refuse contained 46.79 percent carbon and 0.09 percent carbonate carbon. When three other samples of the refuse were heated at 600 C for 2 hours in a muffle furnace, the average percent weight loss was 86.60.

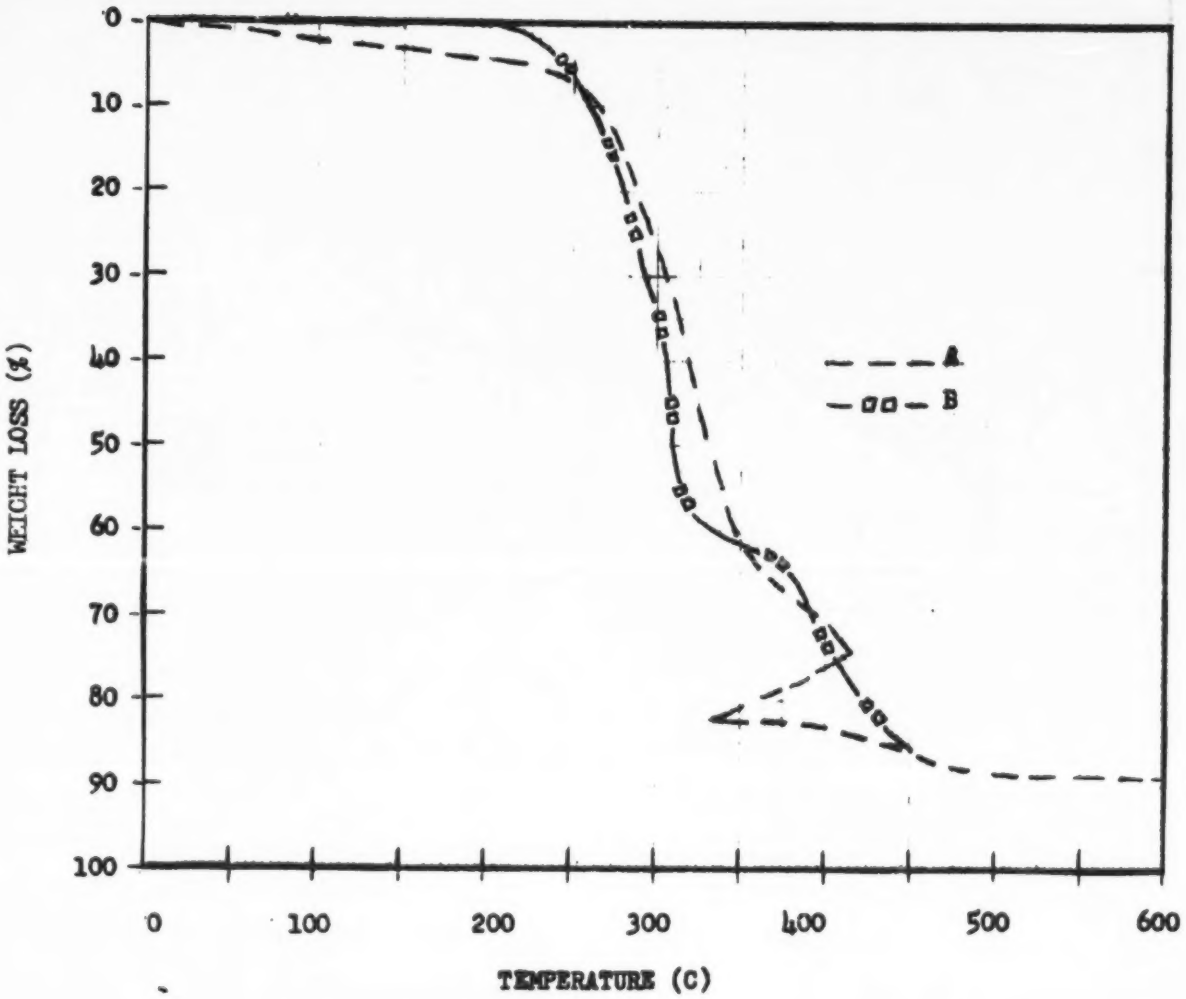

Figure B-2. Percent weight-loss curves of a municipal refuse (RSL 69-5): Effect of heating rate. The curves were derived from thermogravimetric analyses performed by duPont under the following experimental conditions:

	A	B
Sample weight (mg)	9.3	8.3
Atmosphere	Air	Air
Flow rate (liters/hr)	6	6
Heating rate (°C/min)	30	5
Chart speed (in./hr)	18	6

The refuse contained 46.79 percent carbon and 0.09 percent carbonate carbon. When three other samples of the refuse were heated at 600 C for 2 hours in a muffle furnace, the average percent weight loss was 86.60.

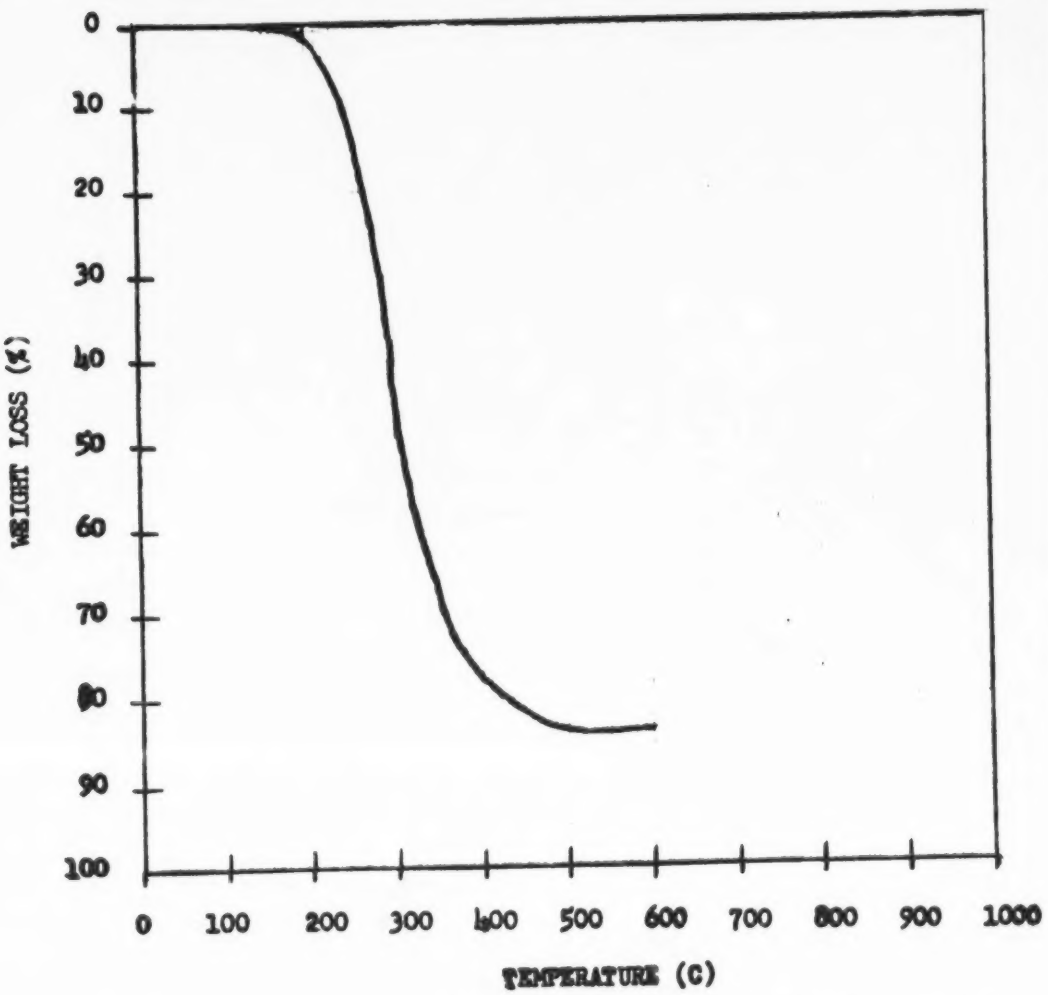

Figure B-3. Percent weight-loss curve of a compost (JC 170-M1). The curve was derived from a thermogravimetric analysis performed by Mettler under the following experimental conditions:

Sample weight (mg)	5.84
Atmosphere	Air
Flow rate (liters/hr)	6
Heating rate (°C/min)	15
Chart speed (in./hr)	12

When a sample of the compost was heated at 600 C for 2 hours in a muffle furnace, the percent weight loss was 86.54.

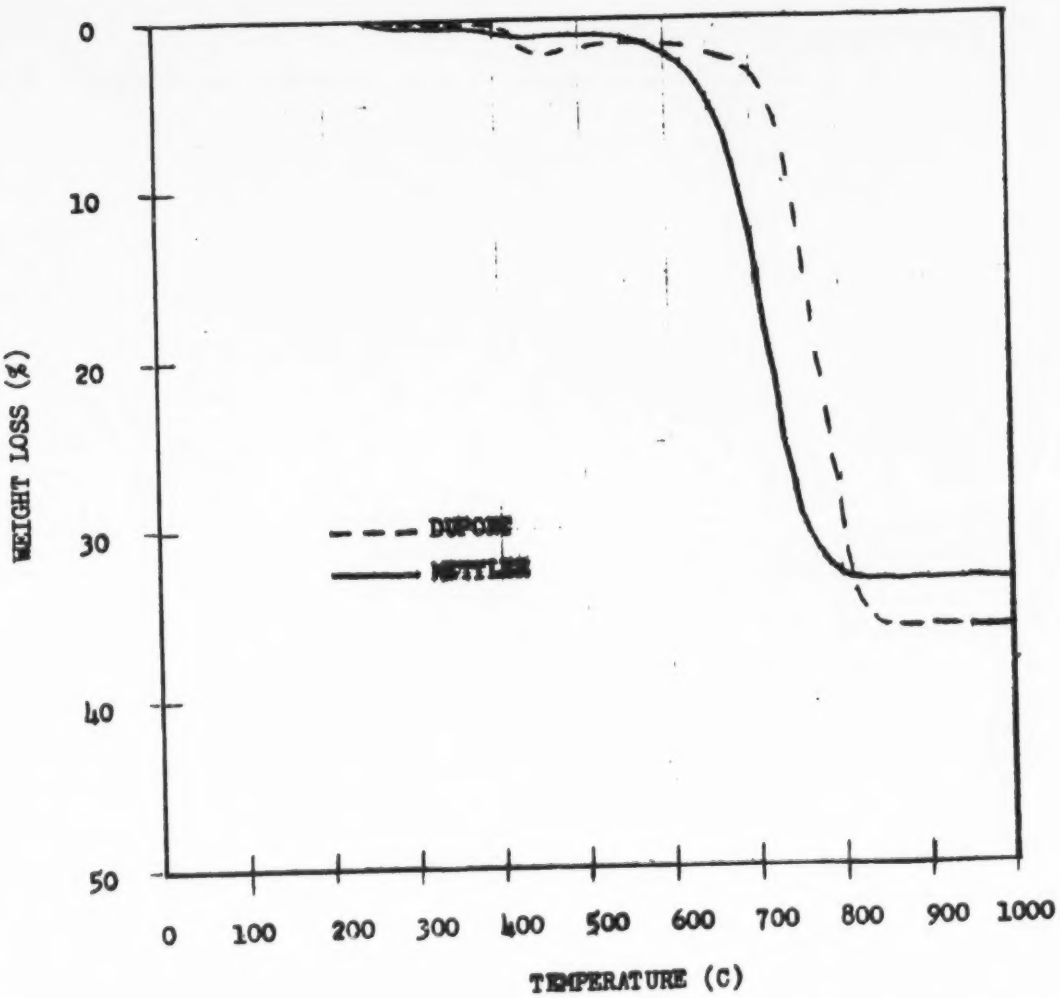

Figure B-4. Percent weight-loss curves of the organic fraction of an incinerator residue (RSL 69-14). The curves were derived from thermogravimetric analyses performed under the following experimental conditions:

	duPont	Mettler
Sample weight (mg)	47	16.75
Atmosphere	Air	Air
Flow rate (liters/hr)	30	6
Heating rate (°C/min)	10	15
Chart speed (in./hr)	6	12

This residue sample contained 8.29 percent carbon and 7.91 percent carbonate carbon. When three other samples of this residue were heated at 600 C for 2 hours in a muffle furnace, the average percent weight loss was 2.16.

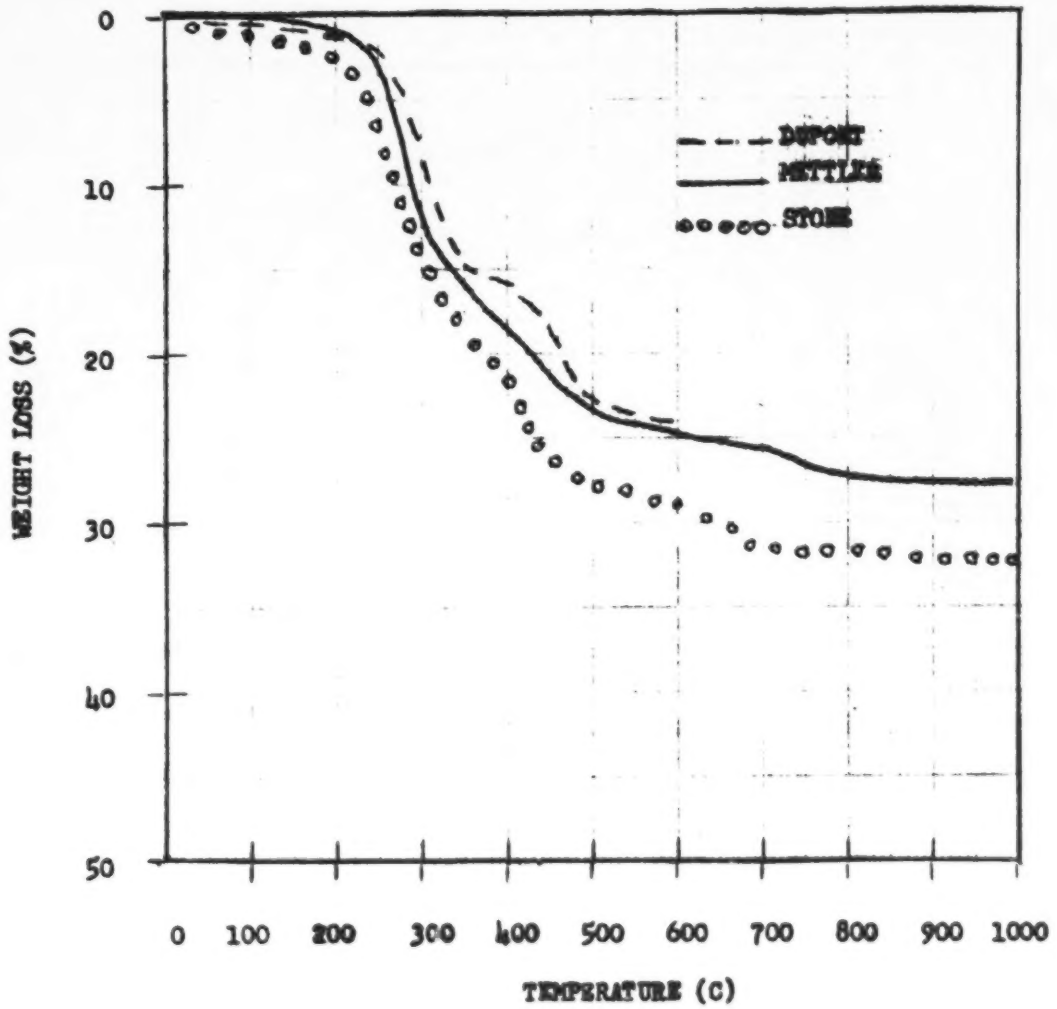

Figure B-5. Percent weight-loss curves of the organic fraction of an incinerator residue (RSL 69-18). The curves were derived from thermogravimetric analyses performed under the following experimental conditions:

	duPont	Mettler	Stone
Sample weight (mg)	24	72.39	100
Atmosphere	Air	Air	Helium
Flow rate (liters/hr)	6	5	1.41
Heating rate (°C/min)	30	4 and 10 at temperatures above 0 and 400 C respectively	10
Chart speed (in./hr)	18	12	5.6

This residue sample contained 12.47 percent carbon and 1.24 percent carbonate carbon. When three other samples of this residue were heated at 600 C for 2 hours in a muffle furnace, the average percent weight loss was 25.21.

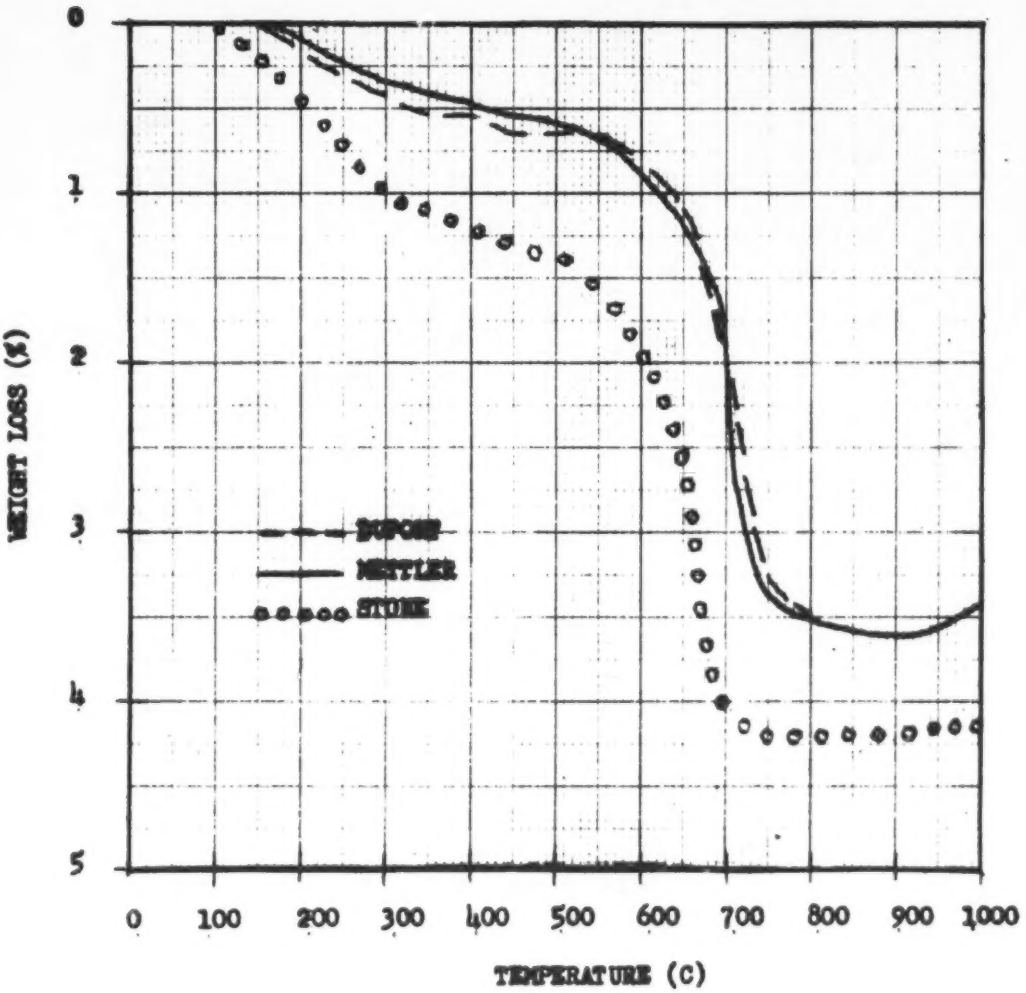

Figure B-6. Percent weight-loss curves of the fines fraction of an incinerator residue (RSL 69-13). The curves were derived from thermogravimetric analyses performed under the following experimental conditions:

	duPont	Mettler	Stone
Sample weight (mg)	49.5	51	200
Atmosphere	Air	Air	Helium
Flow rate (liters/hr)	6	5	1.41
Heating rate (°C/min)	30	10	10
Chart speed (in./hr)	18	12	5.6

This residue sample contained 1.24 percent carbon and 0.58 percent carbonate carbon. When three other samples of this residue were heated at 600 C for 2 hours in a muffle furnace, the average percent weight loss was 0.53.

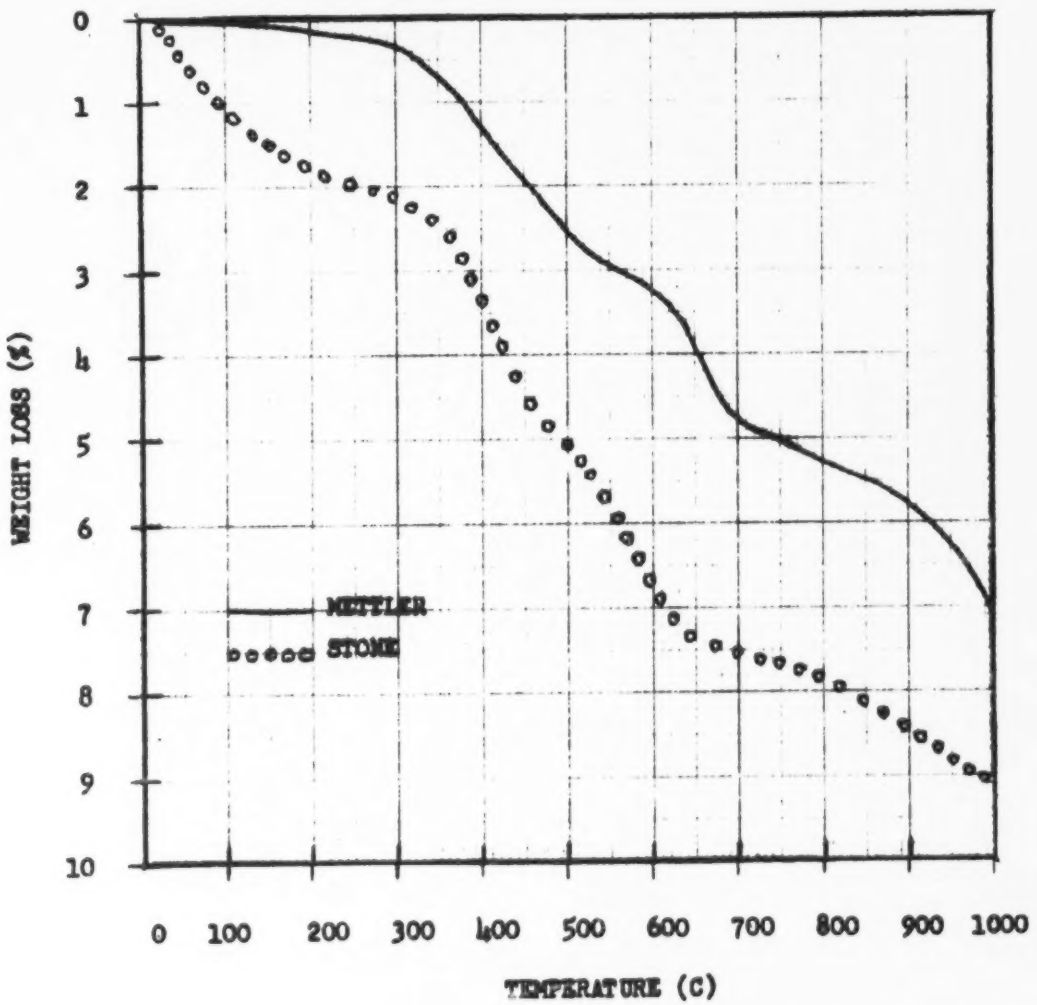

Figure B-7. Percent weight-loss curves of an incinerator fly ash (RRL 69-22). The curves were derived from thermogravimetric analyses performed under the following experimental conditions:

	Mettler	Stone
Sample weight (mg)	88.65	200
Atmosphere	Air	Helium
Flow rate (liters/hr)	5	1.41
Heating rate (°C/min)	4, 6.3, and 10 at temperatures above 0, 400, and 500 C respectively	10
Chart speed (in./hr)	12	5.6

The fly ash contained 3.39 percent carbon and 1.23 percent carbonate carbon. When three other samples of the fly ash were heated at 600 C for 2 hours in a muffle furnace, the average percent weight loss was 3.82.

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